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Interlaboratory Comparison for Analysis of Histamine in Fish as Per ISO/IEC 17043:2010 and ISO 13528:2015

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Abstract

Histamine is a significant food quality, safety, and trade issue in the fisheries sector. In the absence of adequate proficiency testing for histamine in India, it was important to organize a proficiency testing (PT) program among 22 food analytical laboratories for the external assessment of proficiency of the laboratories following ISO 17043:2010 and ISO 13528:2015. The test item (fish) prepared was sufficiently homogenous and stable for the entire duration of the PT program. The Grubbs test employed on the data showed the absence of any outlier result in the present testing scheme. The assigned value of 45.31 mg kg^{-1} was determined using the 'consensus approach' with a standard uncertainty of 1.33 mg kg^{-1}. The robust standard deviation value was 5.02 mg kg^{-1}. The z score for the participant results varied from -1.96 to +1.47, implying that the analytical performance of the participant laboratories was in agreement with the criteria mentioned in ISO 17043:2010. The HorRat value of 1.13, lay between the acceptable range of 0.5-2.0. The distribution of the results as depicted by the kernel density plot revealed a fairly normal distribution of the participants. All the participant laboratories were able to satisfy the proficiency testing requirements, as implied by the z score: -1.96 to +1.47. Further, proficiency testing can be carried out at various analyte levels and other matrices of commercial importance to allow for effective quality assurance.

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Introduction

Globalization, per se, the interdependence among nations due to economic integration arising due to trade and investments has stimulated the economic growth of various countries and has given them economic opportunities in foreign arenas (Surugiu & Surugiu, 2015). This trade liberalization and interconnectedness has created a quality consciousness among traders and consumers. Therefore, the testing centres and laboratories have to demonstrably operate at an internationally acceptable level of competence.

Accreditation offers a unique mechanism for realizing positive assurance, as mentioned in Article 6 of the World Trade Organization's Agreement on Technical Barriers to Trade (TBT) (1994). Through the process of accreditation, an authoritative body is involved in giving a formal stature to an analytical laboratory found competent enough to perform specific analyses. Thus, the accreditation process helps in determining the competence of the laboratory. Competent performance is not only desired by the laboratory itself but also by its clients, as well as regulatory laboratory accreditation agencies and other organizations, involved in specifying requirements of the laboratories.

ISO 17025:2017 is a worldwide accepted standard for accreditation for laboratories involved in testing and calibration and the standard specifies participation in proficiency testing as an essential process requirement for its accreditation. The 'proficiency testing' involves evaluating participants' performance against pre-established criteria employing inter-laboratory comparison. Therefore, it assists in

ensuring the validity of the test results by comparing the analytical performance of a group of laboratories. Proficiency testing trials can be carried out for testing, calibration, and inspection; although the format of schemes may vary according to the sector's requirements, nature of proficiency test items, methods, the number of participants, etc. (ISO 17043:2010).

Histamine, a biogenic amine is produced via enzymatic decarboxylation (microbial decarboxylases) of the free histidine present in the fish tissue. The principal bacteria responsible for the production of histidine decarboxylases are *Enterobacter aerogenes, Morganella morganii, Serratia planticola, Proteus vulgaris etc.* (Duflos et al., 2019). FSSAI (2011) provides a comprehensive list of fish species from 10 families that have the potential to cause histamine poisoning. The bacterial decarboxylation is hastened when harvested fish is subjected to temperatures above 4.4°C at any stage including capture, storage, handling, and processing (US FDA, 2022). Histamine production is rapid at higher temperatures >21°C (Laly, Anupama, Ashok Kumar, & Sankar, 2019) and may not be accompanied by other spoilage signs. Furthermore, even at high histamine levels, the sensory characteristics are seldom affected. It may be noted that cooking may cause inactivation of histamine-producing bacteria but histamine being heat resistant remains intact in food products. The consumption of histamine-containing fish causes histamine fish poisoning (Scombroid poisoning). The effect of histamine poisoning may be slight (8-40 mg), intermediate (40-100 mg), or severe (>100 mg) depending on the amount ingested (Uchoi et al., 2018). The major symptoms of histamine poisoning are swelling of the face and tongue, rashes, oral burning sensation, metallic taste, nausea, diarrhea, vomiting, flushing headache, sweating, dizziness, palpitation, and hypotension (Evangelista et al., 2016). The preventive measure for histamine formation is delaying the growth of histamine formers and simultaneously slowing down the enzymatic activity. Rapid freezing/chilling is mandated after death to arrest the formation of histamine.

Histamine is a significant food safety and trade issue in the fisheries sector. Unacceptable histamine levels render the seafood unfit for sale and therefore cause economic loss to the buyers. There have been multiple instances of histamine-related illness in the past (Demoncheaux et al., 2012; Velut et al., 2019; US FDA, 2019; DeBeer, Bell, Nolte, Arcieri, & Correa, 2021; Pereira et al., 2021). EU's Rapid Alert System for Food and Feed (RASSF) database displays four alerts about the occurrence of histamine in fish exported from India since 2020 (RASSF, 2024). Further, the FDA Freedom of Information Act (FOIA) database lists 53 recalls for histamine in uncooked fish (mostly yellowfin tuna) (DeBeer et al., 2021).

Food Safety and Standards Authority of India (FSSAI) has notified fish species belonging to ten families viz., Carangidae, Chanidae, Clupeidae, Coryphaenidae, Engraulidae, Istiophoridae, Mugilidae, Pristigasteridae, Scombridae, and Xiphiidae, which may cause histamine poisoning. Further, the maximum allowable limit of histamine in various fish products is depicted in Table 1 (FSSAI, 2011). The established levels of histamine or any other contaminant are important from the food safety aspect and have ramifications in the food trade.

Thus, the detection of histamine becomes extremely important from a food safety perspective. Several analytical laboratory methods (spectrophotometry, liquid chromatography, gas chromatography), and quick tests using ELISA kits are available for determining histamine concentration in various fish products. ISO 19343:2017 specifies a method based on high-performance liquid chromatography (HPLC) to determine histamine in fish and fishery products intended for human consumption. Duflos et al. (2019) conducted interlaboratory exercise among nine laboratories for histamine analysis and found the HorRat ratio to be 3.27, 1.63 and 1.36 at 25 mg kg^{-1}, 100 mg kg^{-1} and 220 mg kg^{-1} spiking levels in tuna. They inferred that the method ISO 19343:2017 is consistent for the detection and quantification of histamine in fish and fishery products as per criteria for European Regulation (EC) No. 2073/2005 (European Union, 2005).

The information available on the interlaboratory study for quantitative determination of histamine on fish and fishery products of India is scanty. Therefore, the present study was designed for external assessment of competency of the analytical performance of the laboratory using the proficiency testing approach.

Material and Methods

The entire study was carried out in an in-house

facility accredited with ISO 17025:2017 and none of the activity was subcontracted. Twenty-two laboratories participated in the proficiency trial. The study for histamine quantification was carried out in thermally processed fish muscle and the target concentration was kept at 50 ppm.

The test material sent to the participant laboratories was prepared at ICAR-Central Institute of Fisheries Technology, Proficiency Testing Provider Cell. The tuna fish (*Thunnus albacares*) utilized for test material preparation was cleaned, deboned, and then kept in iced conditions. Certified reference material (CRM) (ISO 17034:2016) of histamine dihydrochloride (Sigma Aldrich, Germany) was used for spiking at 50 mg kg^{-1} level. During all the stages of preparation and spiking, the test material was maintained below 4°C. The test material and CRM were thoroughly blended in a cutter-mixer (Robot-Coupe, USA) to obtain a homogenous material. The fish sample was divided into sub-portions of 30 g each, filled in retort pouches (silicone dioxide/ nylon/ cast polypropylene) (M/s Floeter India Pvt. Ltd., Manesar, Haryana), sealed, and thermally processed to F_0 value of 8 min in an over-pressure retort (Model No. 5682, John Fraser & Sons Ltd., New Castle upon Tyne, U.K.). Thermocouple probes (SSA 12040 G700 TS, Ellab Co. Denmark) were fitted into the core to reach the geometrical centre for monitoring the temperature at the coldest point and the temperature was recorded using thermocouple glands (Model No. GKJ 13009 C042, Ellab Co. Rødovre, Denmark). Samples were then randomly coded to avoid any collusion between the participants. The processed test material along with a blank matrix (prepared similarly) was dispatched by post at ambient temperature. An accompanying letter containing the necessary details and a timeline to be followed was also sent.

The level of histamine in the test item was determined according to the standard method ISO19343:2017 based on HPLC.

A 5-gram portion of the homogenized test item (fish) was extracted in 10 ml of perchloric acid along with 1,7-diaminoheptane (internal standard). The compound 1,7 diamino heptane was used as an internal standard because it resolves nicely from other amines, elutes close to histamine, and is stable (Smith & Davies, 1987; Duflos et al., 2019). The extracted mix was then centrifuged at 8000 g for 5 min at 4°C.

The dansylation (derivatization) of the extracted histamine was carried out using dansyl chloride under alkaline conditions. The supernatant (100 µl), sodium carbonate (300 µl), and dansyl chloride (400 µl) were vortexed and incubated in the dark (60°C, 5 min). Due to the light-labile nature of the dansylated derivative of histamine, the dansylation was carried out in dark conditions. The reaction mixture was then cooled and mixed with 100 µl proline, and then further incubated in the dark at ambient for 15 min. The addition of proline helps in the removal of excess dansyl chloride present in the reaction mixture.

500 µl of toluene was mixed with the reaction mixture. The addition of toluene helps in the extraction of the dansylated histamine from the reaction mixture and the aqueous phase having the dansylated amino acids is discarded. The upper organic phase containing dansylated histamine was dried under nitrogen. The dried residue was suspended in 60% acetonitrile (200 µl) and filtered. It must be noted that histamine solutions should be stored in plastic vials/ containers since it is adsorbed on the glass surface.

The separation of histamine and internal standard was achieved on a C18 column (RP; 250 mm × 4.6 mm × 5 µm; 100 Å Kromasil, Sigma Aldrich; injection vol- 20 µl) with a water/acetonitrile gradient (1ml/min), in a Thermo Scientific UHPLC Dionex Ultimate 3000 equipped with DAD-3000 diode array detector. The detection occurred at 254 nm and chromatograms were observed for histamine and internal standard peaks. The histamine concentration was performed by calculating each response factor against 1,7 diamino heptane and analyzed using the following formula (Equation 1):

$$C_H = \frac{(A_H/A_{IS})\ X\ (5/m)}{a}$$

where C_H is the concentration of histamine in the sample (mg kg^{-1}), A_H is the area of the histamine peak, A_{IS} is the area of the internal standard peak, a is the slope of the calibration curve and m is the mass of the sample.

The homogeneity of the test item was assessed according to ISO 13528:2015 (Annexure B). Ten proficiency test items were randomly selected and analyzed for histamine in duplicates. The test material is regarded as sufficiently homogeneous if the between-sample standard deviation (s_s) is less

than 0.3 times the σ_{PT} (standard deviation for proficiency assessment) (Equation 2) i.e.,

$s_s \leq 0.3 \times SDPA$

This model describes that s_s contributes < 10 % of the variance for evaluation of the performance and therefore evaluation does not change much.

For this, value was determined according to the modified Horwitz model (Thompson, 2000) as described below (Equation 3)

$$\sigma_{PT} = \begin{cases} 0.22 \times c, & \text{when } c < 1.2 \times 10\text{-}7 \\ 0.22 \times c^{0.8495}, & \text{when } 1.2 \times 10^{-7} < c \leq 0.138 \\ 0.01 \times c, & \text{when } c > 0.138 \end{cases}$$

where, c is the mass fraction of histamine, and $0 \leq c \leq 1$. The homogeneity was assessed before dispatching test items to the participants. Stability is assessed after the result submission deadline. A twenty-five-day gap was kept between sample dispatch and the stability test.

The stability of the PT test material was evaluated (ISO 13528:2015) after the deadline for result submission. Three proficiency test items were randomly selected and each item was analyzed in duplicates for histamine content. The test material is considered to be sufficiently stable if the difference between the homogeneity mean and stability mean is less than 0.3 times the σ_{PT} (calculated using the Horwitz approach) (Equation 4) i.e.,

$$|\overline{y}_1 - \overline{y}_2| \leq 0.3 \times \sigma_{PT}$$

The statistical evaluation for the results obtained from participant laboratories was carried out according to ISO 13528:2015 which describes the statistical methods for proficiency testing providers while designing PT schemes and analyzing the data therefrom. It provides the support for implementing ISO 17043. The consensus value, uncertainty of the consensus value, standard deviation of the proficiency assessment, and z-scores were calculated as described in the standard. Initially, Grubb's test was employed to identify the outliers among the observations.

The assigned value (AV) (X_{PT}) for the proficiency test item as robust sample average (X*), and robust standard deviation (s*) were determined by the consensus approach from the participant results, using the robust analysis: Algorithm A-iterated scale (ISO 13528:2015, Annexure C).

The validation of AV was conducted by comparing the consensus value with the reference value. For a valid AV, the difference should be more than twice its standard uncertainty. The standard uncertainty of the AV was calculated by following the formula (Equation 5)-

$$u(X_{PT}) = 1.25 \times \frac{s^*}{\sqrt{p}}$$

where s* is the robust standard deviation of the results (standard deviation- proficiency assessment) and p is the number of participant laboratories. In this equation, the uncertainty of AV is assumed to encompass the uncertainty effects due to inhomogeneity, instability, and transport. The uncertainty estimation further validates the AV in the PT scheme. The criterion for validating the AV is that the uncertainty calculated should be less than 0.3 times the SDPA (Equation 6) i.e.,

$$u(X_{PT}) \leq 0.3 \times \sigma_{PT}$$

Further, the assigned value was compared with the reference value to find out bias, and the standard uncertainty of the difference ($X_{ref} - X_{PT}$) was calculated employing the following formula (Equation 7):

$$u_{diff} = \sqrt{u^2 (X_{ref}) + u^2 (X_{PT})}$$

where $u(x_{ref})$ is the uncertainty of the reference value and $u(x_{PT})$ is the uncertainty of the AV.

The performance of the laboratories was evaluated employing the z scores. The z score for individual result X_i was calculated using the following model (Equation 8)–

$$z = \frac{(X_i - X_{PT})}{\sigma_{PT}}$$

where X_{PT} is the AV and σ_{PT} is the standard deviation of participant results. The z-scores were interpreted according to the guidelines of ISO 13528:2015 ($|z| \leq 2.0$- acceptable result, $2.0 < |z| < 3.0$- warning signal, $|z| > 3.0$- unacceptable result). The z-score of the participants' results is plotted as a bar chart for visualization (Fig. 1)

Results and Discussion

The measurement of homogeneity and stability ensures that every participant receives a comparable

Table 1. Limits of histamine level in fish and−fishery products

Sl. No.	Product category	Applicable	Histamine level n	c	m (mg kg^{-1})	M (mg kg^{-1})
1	Raw/ chilled/ frozen finfish	Species with high amounts of free histidine (listed fish species with potential to cause histamine fish poisoning)	9	2	100	200
2	Thermally processedfishery products		9	2	100	200
3	Smoked fishery products		9	2	100	200
4	Fish mince/ surimi and analogues		9	2	100	200
5	Battered and breadedfishery products		9	2	100	200
6	Other ready-to-eat fisheryproducts		9	2	100	200
7	Other value-added fisheryproducts		9	2	100	200
8	Other fish-based products		9	2	100	200
9	Dried/ salted and driedfishery products		9	2	200	400
10	Fermented fishery products		9	2	200	400
11	Fish Pickle		9	2	200	400

(Adapted from FSSAI, 2011) (n: no. of units from the sample; c: Max. allowable no. of unacceptable samples; m: acceptable level in a sample; M: specified level when exceeded in one or more samples would cause the lot to be rejected)

test item, which remains stable with no significant changes during storage and transportation throughout proficiency testing. The assessment of performance shouldn't be negatively impacted by the test matrix's inhomogeneity or instability. S_s value (0.76) was less than 0.3 times σ_{PT} (1.33) indicating homogeneity in the proficiency test items prepared in the laboratory. The difference between the homogeneity mean and stability mean (0.27) was less than 0.3 times σ_{PT} (1.33) indicating the stability of the test item for the entire duration under the availed transportation conditions.

The summary statistics for the results obtained from the proficiency testing trial conducted are depicted in Table 2. A total of 22 laboratories participated in the proficiency testing program.

The limit of quantification for histamine analysis for various laboratories ranged from 5-25 mg kg^{-1}. The evaluation of the laboratories' performance is done by comparing their results with a common result value called an assigned value (AV). Before identifying the assigned value, the blunders are removed from the result data set by visual reviewing through histograms and other statistical tools. Outlier tests are further employed to support the visual review for aberrations and help in rejecting the outliers. The various outlier rejection tools applicable to inter-laboratory data are mentioned in ISO 16269-4:2010 and ISO 5725-2:2019. In the present study, Grubb's test for outliers was employed for identifying the extreme values. The test is generally performed when the number of outliers is small (1-2 outliers) and it considers the standard deviation of all the participants including all the potential outliers. The

Table 2. Summary statistics for proficiency testing of histamine analysis in fish

Description	Value
Number of participants	22
Outlier results	nil
Assigned value (mg kg^{-1})	45.31
Robust standard deviation (mg kg^{-1})	5.02
Standard uncertainty (mg kg^{-1})	1.33
x_{diff} (mg kg^{-1})	4.69
u_{diff} (mg kg^{-1})	2.61
Skewness	-0.43
Kurtosis	-0.42
HorRat value	1.13
Proficiency evaluation	z score
z score range	-1.96 to +1.47

Grubbs test value (G_{test}) was observed to be less than the critical G value ($G_{critical}$; α- 0.05) (2.08 < 2.76), implying the absence of outliers in the test results.

The assigned value can be determined by five methods viz., formulation, certified reference material, results from a single laboratory, consensus value from expert laboratories, and consensus values from participant results (ISO 17043, ISO 13528). Since the participants were allowed to choose their measurement approach, the consensus approach was utilized to determine the AV. The usage of the consensus approach for AV determination gives additional confidence to the participant laboratories. The AV for histamine was 45.31 mg kg^{-1} and the robust standard deviation as obtained by Algorithm A with iterated approach was 5.02 mg kg^{-1}. The consensus approach helps in easy and cheap measurement of the assigned value of the measurand (histamine). Sometimes, it may act as the only method that can be employed for the estimation of true value with natural matrices. The standard uncertainty of the assigned value was 1.33 mg kg^{-1} and it was in agreement with the criteria mentioned in ISO 13528:2015 (1.33 < 1.51); thus, the uncertainty is not required to be taken into account while interpreting the results of the proficiency tests. Further, this means that the AV was precisely estimated. The comparison with the reference value (x_{diff}) further indicated that the assigned value was rightfully determined. Both the participant outcomes and the reference measurement technique are free of bias and the proficiency testing scheme is fine.

The skewness is a measure of the symmetry of data while the kurtosis determines the flatness of the curve (or convexity). The skewness and kurtosis value for the results were obtained to be -0.42 and -0.43, respectively signifying that the data set is symmetrical and mesokurtic to a greater extent.

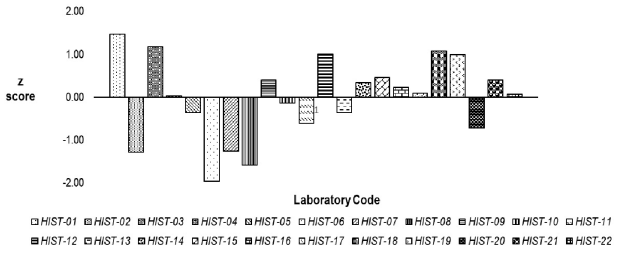

Fig. 1. Bar chart showing z- scores of the proficiency testing participants for histamine analysis in fish

The HorRat value is a performance metric displaying the acceptability of analytical methods for among-laboratory precision (reproducibility). It is the ratio of the observed relative standard deviation among laboratories and the predicted relative standard deviation (Horwitz model). This value is unaffected by the analyte, matrix, and method. HorRat value defines the closeness between the method precision (reproducibility) and predicted value and equals 1 for exact correspondence (Horwitz & Albert, 2006) and its acceptable range is from 0.5 to 2.0. The HorRat value (1.13) was well within the acceptable range. It is noted that values lower than 0.5 may show underreported average or superior experience and training while values

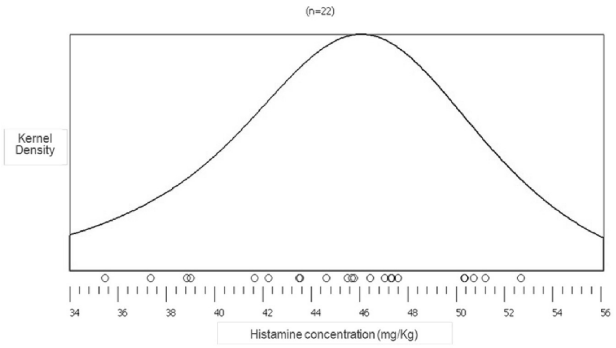

Fig. 2. Kernel density plot depicting the distribution of the participant results for histamine analysis in fish

higher than 2.0 indicate inhomogeneity in samples, unsatisfactory method and/or operation (Horwitz & Albert, 2006).

The performance of the participant laboratories, assessed using z-scores (-3.00 to +3.00) are plotted using a bar chart for better visualization (Fig. 1). The bar chart presents the performance score in a single graph and might help in revealing any commonality in the scores. For z scores, a limit of 2.0 and 3.0 is taken as a warning signal or action signal. It is assumed that the measurements performed correctly are described by a normal distribution (mean: x_{PT} ; standard deviation: σ_{PT}) and then z scores generated are normally distributed (mean- 0; standard deviation- 1.0). Under these conditions, the possibility of the z score going beyond the range ($-3.0 \leq z \leq +3.0$) could occur due to major aberration rather than chance and, there is a need to scrutinize the laboratory performance. The z scores for the

participants varied from -1.96 to +1.47 and could not generate a warning/ action signal. Thus, there is no need to review the laboratory performance. In general terms, some of the factors responsible for undermining the laboratory performance could be classified as clerical (mislabeling, reporting, transcription error), technical (measurement procedure, training, storage of PT item, environmental conditions, data processing, measurement procedure, equipments, reagents) and problem related to PT scheme (sample homogeneity/ stability, matrix difference between PT item and routine test items, inappropriate assigned value/ SDPA, level outside scope).

Further, the data was represented using a kernel density plot for better visualization. A kernel density plot curve describes the general pattern of the distribution and is generally used for moderate to large data sets. The kernel density curve's shape is used to determine the distribution from which the data were collected, and distinct modes emerge as different peaks.

Similarly, the outlier values appear as separate peaks separated from the remaining data. The distribution of the results as depicted by the kernel density plot revealed a fairly normal distribution of the participants (Fig. 2).

An independent assessment of the technical competence of the laboratory generates the confidence necessary for realizing the genuineness of test outcomes. Proficiency testing offers independent assessment by objectively comparing the test results of various laboratories for the desired analyte. In the present study, all the participant laboratories were able to satisfy the proficiency testing (ISO 17043:2010) requirements, as implied by the z score-1.96 to + 1.47. The results obtained could be used to improve the overall effectiveness and performance of the laboratory. As emphasized by Brookman and Mann (2021), the focus of proficiency testing (PT) should be on learning from the results rather than merely on passing or failing. Further, proficiency testing can be carried out at various analyte levels and other matrices of commercial importance to allow for effective quality assurance. A thoroughly competent laboratory could act as a wonderful tool for effective surveillance, supply chain traceability, and identifying supply gaps among various other food safety interventions, thus assuring food safety for the public.

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